Enhanced in-line detection, cleaning and repair of nano scale defects in thin films for flexible photovoltaic and food packaging applications

A European collaboration that brought together world class expertise from industry, technology and academia
Introduction

NanoMend was an industry-led collaboration where the primary aim was to develop pioneering technologies for in-line detection, cleaning and repair of micro and nano-scale defects on thin films used in applications for flexible photovoltaics and paper-based food packaging films. The project ran from January 2012 to December 2015 and comprised of 14 partners across 6 European countries.

Further aims for the project included demonstrating the ability to implement roll to roll atomic layer deposition (ALD) coating technologies to produce ultra barrier coatings for both flexible photovoltaic (PV) modules and polymer coated paper. To test the functionality of the barriers a traceable water vapour transmission rate (WVTR) instrument was built in the consortium, allowing the lowest detectable WVTR’s to be traceably measured.

Early experimental work in the project led to a deeper understanding of the nature of substrate and thin coating layer defects in both the flexible PV and polymer coated paper products. From this research defect classification systems were developed which led the research teams to understand the size and morphology of critical substrate defects, which were detrimental to the function of the PV and paper based films. By understanding and targeting the most critical defects for the substrates under study the consortium were able to design specify and develop two technology demonstrator systems for the PV and Paper industries and a further two proof of concept measurement systems for direct measurement of ALD Al2O3 coatings and embossed holographic film.

Going forward it is predicted that the demonstrator capabilities will be seen to have significant advantages in their application sectors and as such, be implemented into full-scale manufacturing lines, thus facilitating significant manufacturing efficiency gains for end user industries. Additionally the technology suppliers within the consortium now have the know how to address significant new markets in coated paper and printed electronics in the area of defect detection and substrate cleaning.

Professor Liam Blunt, Director of the Centre for Precision Technology at the University of Huddersfield and Project Co-ordinator for NanoMend.
Nanomend Achievements

The project has several significant achievements:

- Development of the world first traceable WVTR facility with capability down to < $5 \times 10^{-5}$ g/m$^2$/day.
- Modelling of WVTR through thin film coatings.
- Implementation of a wavelength scanning interferometry for in-process measurement of barrier film defects.
- Development of two in-line camera based systems for defect detection in flexible PV and coated paper manufacturer.
- Development and demonstration of in-line localised cleaning capabilities for both PV and paper manufacturing sectors.
- Licencing agreement and market launch of WSI technologies for in-line metrology.
- Manufacture of single layer ALD ALOx ultra barrier films with demonstrateable WVTR < $1-10^{-4}$ g/m$^2$/day.
- Incorporation of ultra-barrier coating into fully functional front sheet encapsulation for flexible PV.
- Implemented ALD coatings on holographic coated paper products.
NanoMend Consortium

1. University of Huddersfield – Huddersfield, UK
2. National Physical Laboratory – Teddington, UK
3. Fraunhofer-Gesellschaft – Munich, Germany
4. KITE Innovation – Huddersfield, UK
5. CPI – Sedgefield, UK
6. Lappeenranta University of Technology – Lappeenranta, Finland
7. Stora Enso – Helsinki, Finland
8. TNO – Eindhoven, The Netherlands
9. Tampere University of Technology – Tampere, Finland
10. ISRA VISION AG – Darmstadt, Germany
11. ISOVOLTAIC AG – Lebring, Austria
12. Flisom – Duebendorf, Switzerland
13. Iscent Oy – Vantaa, Finland
14. IBS Precision Engineering – Eindhoven, The Netherlands
Technologies Developed by NanoMend

The Need for Defect Detection in Thin Film Manufacturing

Defects can occur at a number of different stages in the manufacturing process and can be caused by anomalies, such as contamination and thickness variations in the film. For example, the occurrence of micro and nano-scale defects in barrier films can allow water vapour to enter flexible solar modules, which is highly detrimental to their performance and lifetimes. Defects also allow gases to enter or leave food packages, which significantly impacts product shelf life. Most thin film coatings for industry are produced at high speed using roll-to-roll manufacturing techniques. These systems require specialist high speed and high resolution equipment to detect and correct any defects in production.

NanoMend has developed seven technology systems that will aid the commercialisation of flexible photovoltaics and aseptic packaging. The project has assessed the economic feasibility of these technologies using roll to roll processes at pilot production scale.

The technologies developed are:

1. Atomic layer deposition for photovoltaics.
2. Atomic layer deposition for aseptic packaging.
4. High sensitivity water vapour transmission rate testing of ultra barrier films.
5. Fourier imaging systems for the in-line inspection of structured surfaces.
7. High-speed high-resolution surface inspection.
The Consortium

University of Huddersfield, UK - The University of Huddersfield worked in conjunction with KITE innovation as co-coordinators for the project in addition to leading the defect detection and characterisation work package. The University of Huddersfield also developed characterisation technique alongside optical sensor development.

National Physical Laboratory, UK - NPL worked on the development of a new fully traceable high resolution WVTR instrument. Additionally the researchers developed a prototype system for surface inspection of holographic surface structures inspection.

Fraunhofer-Gesellschaft, Germany - Fraunhofer led the studies for the analysis, cataloguing and benchmarking of defects in addition to the measurement of WVTR of barrier layers and accelerated lifetime testing. Fraunhofer also worked on measuring the efficiency of rigid CIGS modules.

KITE Innovation, UK - KITE Innovation provided a range of services in the successful project management and exploitation of NanoMend.

The Centre for Process Innovation (CPI), UK - CPI provided expertise and atomic layer deposition samples in a number of technical areas related to the development of barrier film and the detection and characterisation of defects. CPI also delivered the dissemination and exploitation of the project.

Lappeenranta University of Technology, Finland - LUT provided expertise on thin film deposition and surface modification for atomic layer deposition technologies used for packaging applications.

Stora Enso, Finland - Stora Enso's role as an end user in the project was to provide manufacturing knowledge and characterise defects in fibre based packaging in a production scale environment.

TNO, The Netherlands - TNO worked in the development of novel local cleaning and repair strategies alongside supporting the integration of such technologies in the pilot lines.

Tampere University of Technology, Finland - TUT worked on the inspection, cleaning and repair of defects and the integration of these systems into a pilot scale paper packaging line.

ISRA VISION AG, Germany - ISRA VISION designed, developed and integrated a number of high-speed and high resolution inspection systems for the pilot lines in the project. These systems were applicable for the inspection of surfaces on photovoltaic modules and coated paper.

ISOVOLTAIC, Austria - ISOVOLTAIC provided improved transparent multilayer barrier materials for the encapsulation of flexible thin-film solar cells.

Flisom, Switzerland - Flisom was an end user in the project, hosting the project’s defect detection system within their PV pilot manufacturing line. The company provided PV samples for analysis, detection and repair and also guidance in their specification needs and supply of PV modules for analysis, defect detection, and repair.

Iscent Oy, Finland - Iscent Oy demonstrated holographic structures on paper technology in their industrial process. Their role was also to provide a series of tests structures to measure and analyse defects.

IBS Engineering, The Netherlands - IBS Engineering integrated a fully characterised and traceable high resolution wavelength scanning instrument into a pilot line at CPI, enabling measurement across the full web width. It also developed a faster, smaller instrument. The company carried out system integration of the cleaning and inspection systems for the PV demonstrator line.
Technologies Developed by NanoMend:

1. Atomic Layer Deposition for Photovoltaics

Atomic Layer Deposition based thin films are conformal and pinhole-free and therefore ideally suited for demanding thin film applications. ALD can be applied to a wide range of products in order to improve their quality, increase yield, reduce manufacturing wastage and reduce manufacturing costs. ISOVOLTAIC and CPI looked at ways to apply atomic layer deposition technologies to act as ultra barriers in photovoltaic applications.

The following specifications were achieved:

<table>
<thead>
<tr>
<th>Properties</th>
<th>Test Method</th>
<th>Unit</th>
<th>Value</th>
<th>C.O.A</th>
</tr>
</thead>
<tbody>
<tr>
<td>WVTR</td>
<td>NPI, (9h+90K, T=35°C)</td>
<td>g/m²/d</td>
<td>≤10⁻⁴</td>
<td>x</td>
</tr>
<tr>
<td>Thickness</td>
<td>Internal Method</td>
<td>mm</td>
<td>0.125 ± 0.10%</td>
<td>x</td>
</tr>
<tr>
<td>Mass Per Unit Area</td>
<td>Internal Method</td>
<td>g/m²</td>
<td>177 ±10%</td>
<td>x</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>MD</td>
<td>DIN 53455/ASTM-D-882</td>
<td>N/mm² or MPa</td>
<td>≥155</td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>TD</td>
<td>DIN 53455/ASTM-D-882</td>
<td>N/mm² or MPa</td>
<td>≥155</td>
</tr>
<tr>
<td>Shrinkage (30 min at 150°C)</td>
<td>MD</td>
<td>Internal Method</td>
<td>%</td>
<td>≤0.2%</td>
</tr>
<tr>
<td>Transmission at 450 - 800nm</td>
<td>TD</td>
<td>Internal Method</td>
<td>%</td>
<td>≥0.15%</td>
</tr>
<tr>
<td>UV (30000h) and DH (1000h)</td>
<td>None</td>
<td>Yellowing index / L-a-b Values</td>
<td>&lt;5</td>
<td>-</td>
</tr>
</tbody>
</table>

2. Atomic Layer Deposition for Aseptic Packaging

The objective for the application of ALD is to increase the shelf life of packaging through improving the performance and yield of thin barrier layers. The technology also seeks to reduce material wastage during production.

Lappeenranta University of Technology, Tampere University of Technology and Stora Enso worked in collaboration on this technology.

The following specifications were achieved:

With 30nm thick Al2O3 coating, O2 TR of BOPP film was decreased from 1100cm³/m²/24h down to 20cm³/m²/24h at 23°C, 0%RH.

3. In-line ultra precision 3D surface metrology for roll-to-roll thin film processing utilising Wavelength Scanning Interferometry (WSI)

CPI, IBS Precision Engineering and the University of Huddersfield developed a new optical interferometry system for fast in line areal surface measurements. With WSI, micro and nanoscale surfaces, defects can be measured, where environmental noise is compensated within the measurement system. The WSI system has been implemented at CPI as a demonstrator sensor for the detection of defects in polymer film. This will be a showcase of the fast, large range, nanometre resolution and robust capabilities of the WSI.

The WSI technology is protected by pending patents derived from PCT/GB2010/050063 also published as US2012026508(A1).
The following specifications were achieved:

<table>
<thead>
<tr>
<th>WSI specifications</th>
<th>Value</th>
<th>unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertical range (lens dependent)</td>
<td>96 (2X)</td>
<td>μm</td>
</tr>
<tr>
<td>Lateral range (lens dependent)</td>
<td>2.8x2.8 (2X)</td>
<td>mm</td>
</tr>
<tr>
<td>Pixels over FOV</td>
<td>1000 x 1000</td>
<td>pixel</td>
</tr>
<tr>
<td>Vertical resolution</td>
<td>2</td>
<td>nm</td>
</tr>
<tr>
<td>Measurement time (typically)</td>
<td>&lt;1</td>
<td>sec</td>
</tr>
<tr>
<td>Stabilisation bandwidth</td>
<td>&lt;500</td>
<td>Hz</td>
</tr>
</tbody>
</table>

4. High Sensitivity Water Vapour Transmission Rate (WVTR) Testing of Ultra Barrier Films

Many of the materials used in the production of electronic products are highly chemically sensitive and will react with a wide range of environmental components. Photovoltaic modules and flexible displays are particularly sensitive in this regard as they require the use of low work function metal cathodes which are extremely sensitive to water.

CPI and the National Physical Laboratory collaborated in the development of two complimentary high sensitivity WVTR techniques for the testing of Atomic Layer Deposition barriers. NPL’s is highly accurate and traceable to their dynamic water standard and CPI’s is high throughput and low cost.

The following specifications were achieved:

<table>
<thead>
<tr>
<th>Product Information</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lower Detection Limit</td>
<td>3 x 10⁻⁵</td>
<td>g m⁻² day⁻¹</td>
</tr>
<tr>
<td>Temperature Range</td>
<td>22–36</td>
<td>°C</td>
</tr>
<tr>
<td>Controlled RH Testing</td>
<td>50 to 90</td>
<td>%</td>
</tr>
<tr>
<td>Sample Size</td>
<td>100 x 100</td>
<td>mm</td>
</tr>
</tbody>
</table>

CPI’s parallel test is capable of high temperature ranges (e.g. 100°C). Parallel testing enables high throughput and the sample area can be tailored to the application.
5. Fourier imaging systems for the in-line inspection of structured surfaces

The scattering of light from periodic micro-structures on the surface of plastic films is the basis of the optical appearance found in, for example, premium gift-wrap and anti-counterfeit film on high-value toiletries. These optical grating structures are typically produced in high volume using roll-to-roll processes such as thermomechanical embossing. Adaptable, high-speed and quantitative verification of such optical grating structures over vast regions is both slow and prohibitively expensive to achieve with conventional instruments due to the high dynamic range challenge. Currently, a sparsely sampled global inspection ‘by eye’ is employed, which is highly subjective and suffers from poor repeatability. To reimagine the metrology solution, the National Physical Laboratory and Fraunhofer are developing and applying a variety of Fourier imaging techniques for the affordable inspection of these optical gratings.

Fourier imaging systems enable rapid quality mapping of optical grating structures that is as intuitive as inspection by eye, yet faster and more repeatable. The Fourier imaging systems developed in NanoMend can be tailored toward specific performance, cost and compatibility requirements, by the substitution of key components. The systems are easily scalable for monitoring larger substrates in-situ, via parallelisation or cross-web scanning. Alternatively, the systems may be implemented in the user’s offline inspection.

6. Local Cleaning Method for Micron-sized Particle Contamination in Thin Film Processing

TNO and IBS Precision Engineering have collaborated together in the development of a cleaning method to locally remove particles in thin film processing. The low cost, modular cleaning unit which has been developed in the NanoMend project enables the removal of more than 90% of micron sized particles and significantly reduces the occurrence of electrical shunts in the roll-to-roll manufacturing of photovoltaic and of leaks in aseptic packaging.
Developed as part of the NanoMend project, prototypes of the cleaning units have been demonstrated on thin film photovoltaic and paperboard packaging substrates. The photovoltaic unit is based for testing purposes at Flisom’s R&D line in Zurich, Switzerland and the packaging unit located at Tampere University of Technology in Tampere, Finland.

The units have the following specifications:

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Typical Web Speed:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Photovoltaics</td>
<td>1-10</td>
<td>m/min</td>
</tr>
<tr>
<td>Paper Packaging</td>
<td>20-400</td>
<td>m/min</td>
</tr>
<tr>
<td>Local Cleaning Spot Diameter</td>
<td>20</td>
<td>mm</td>
</tr>
<tr>
<td>Web Width</td>
<td>50</td>
<td>cm</td>
</tr>
</tbody>
</table>

**7. High-speed high-resolution surface inspection**

ISRA VISION AG has cooperated with universities and research institutes like TUT, NPL and Fraunhofer in order to develop in-line surface inspection techniques for high-speed high-resolution roll-to-roll applications. These techniques are on the edge on what is possible with optical inspection today and as part of NanoMend were integrated into inspection systems for different sample applications.

**Key Innovative Features:**

The surface inspection systems have three main features:

- Data processing performance of more than 640MB/s image data in one channel. ISRA has further developed the processing speed to 1.2 GB/s.
- Advanced multi-modal lighting techniques.
- Very high resolution down to 3µm for large surfaces.

The key advantage of the systems is the possibility to integrate these techniques towards high-end in-line inspection systems at an economical price.

The system has the following specifications:

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Data processing performance</td>
<td>&gt;650</td>
<td>MB/s</td>
</tr>
<tr>
<td>Resolution</td>
<td>≥3</td>
<td>µm</td>
</tr>
<tr>
<td>Illumination Modes</td>
<td>≤5</td>
<td></td>
</tr>
</tbody>
</table>
Contact:

**Project Co-ordinator**
Liam Blunt
University of Huddersfield

**Marketing Contact**
Steven Bagshaw
CPI

[info@nanomend.eu](mailto:info@nanomend.eu)
[www.nanomend.eu](http://www.nanomend.eu)

---

This project has received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under grant agreement no 280581